#### **CHAPTER V**

# CONTROL OF REACTIVE DISTILLATION COLUMN

In the previous chapter, the optimum configuration of a reactive distillation column for the production of butyl acetate from dilute acetic acid (80 wt %) at steady state conditions has been established. In this chapter, three control structures are designed, which is based on the approach presented in Appendix B, for controlling the reactive distillation column at desired steady state conditions. Dynamic simulation of the reactive distillation is performed based on the determination of the size of the column, i.e., column diameter (2.5 m) and volume of reflux drum (5 m³) and reboiler (7 m³). Detail on the sizing of equipment is further given in Appendix C.

# 5.1 Criteria for selection of best temperature control tray

Many industrial columns use temperatures for composition control because direct composition analyzers can be expensive and unreliable. Controlling these compositions directly requires that we have composition analyzers to measure them. Instead of doing this, it is often possible to achieve fairly good product quality control by controlling the temperature on some tray in the column and keeping one manipulated variable constant. The typical procedure for selecting which tray to control is to look at the steady-state temperature profile in the column at the base-case conditions, as illustrated in Figure 5-1. We look for a location in the column where there are large temperature changes from tray to tray. Then we select the tray where the temperature profile is changing the most from tray to tray.

Other criteria are also commonly used to guide the selection of the control tray.

A first criterion is to find the control tray that is most sensitive to changes in the manipulated variable. Following this guideline, a steady-state rating program is used

to make small open loop changes in the manipulated variable (heat input in this study). We look at the resulting changes in tray temperatures and select the tray that shows the largest change. In Figure 5-1, tray 11 to 24 appears to be the location where changes in heat input (reboiler duty) produce the largest changes in tray temperature. This procedure gives the largest steady-state gain between the controlled variable and manipulated variable. A second criterion is to find the control tray where the steady-state responses are similar for both positive and negative changes in the manipulated variable. This procedure attempts to avoid problems with nonlinearity. Figure 5-1 shows that tray 24 does a good job in satisfying this criterion. So, the temperature at 24<sup>th</sup> stage is selected as the controlled variable of the reactive distillation fed by 80 wt % HOAc.

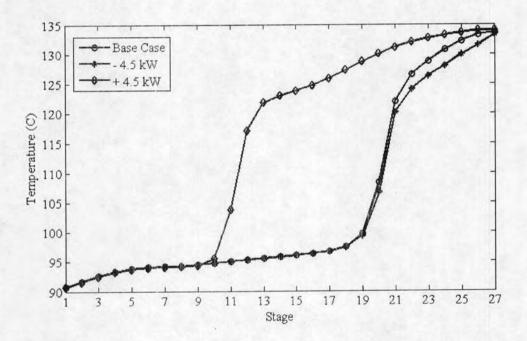


Figure 5-1 Effect of heat duty at reboiler on the column temperature.

#### 5.2 Disturbances

A disturbance upsets the process system and causes the output variables to move from their desired set point. Disturbance variables cannot be controlled or manipulated by the process engineer. The control structure should account for all disturbances that can significantly affect on process. The disturbances to a process can either be measured or unmeasured. In all of the control schemes given in the subsequent section, the following disturbances are assumed:

- Step changes of ± 10 % in the flow-controlled fresh feeds (HOAc and BuOH feed flowrate)
- Step changes in both of HOAc and BuOH composition of the feeds.

## 5.3 Control Structure 1 (CS1)

The typical control structure of reactive distillation is show in Figure 5-2. The fresh feeds of HOAc and n-BuOH are flow-controlled. The column pressure is controlled by manipulating the condenser heat removal. The base level and reflux drum level are controlled by bottom flowrate and distillate flowrate, respectively. The organic reflux flow is flow-controlled. Temperature control of stage 24<sup>th</sup> is implemented by manipulating the reboiler duty. In the control system of the reactive distillation column, the level, pressure, and flow controls constitute inventory control, maintaining the basic operation of the column.

The tuning parameters of the controllers are shown in Table 5-1. Proportional-only level controllers are used. The PI controllers are used in flow and pressure loops. The PID controller is used in temperature loop. In this loop, two first-order measurement lag with a time constant of 30s each are used. All valves are designed to be half open at steady state.

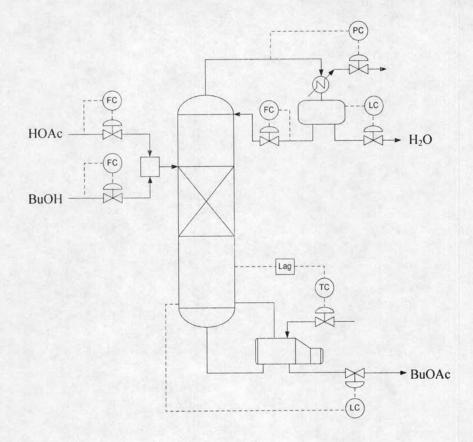


Figure 5-2 Control structure 1: constant reflux flow.

Table 5-1 Tuning parameters for CS1

Temperature controller	$K_c = 2.14$ $\tau_i = 2.73 \text{ min}$
	$\tau_{\rm d} = 0.606 \text{ min}$ $K_{\rm c} = 2.14$
Pressure controller	$\tau_i = 10 \text{ min}$
Level controllers (Reflux drum and Base level)	$K_c = 2$
Organic reflux flow controller	$K_c = 0.5$ $\tau_i = 0.3 \text{ min}$
HOAc fresh feed controller	$K_c = 0.8$ $\tau_i = 0.1 \text{ min}$
BuOH fresh feed controller	$K_c = 0.45$ $\tau_i = 0.3 \text{ min}$

# 5.3.1 Responses of Control Structure 1 (CS1)

In order to evaluate the control performance, the designed control structure is tested under disturbance condition. First, the step changes of  $\pm$  10 % fresh feeds (HOAc and BuOH) are used as disturbances of the process. These responses are shown in Figures 5-3 and 5-4. The responses of the step changes in feeds composition (HOAc and BuOH) are shown in Figure 5-5.

When the flow rate of HOAc is increased by 10%, it can be from Figure 5-3 that CS1 is able to maintain temperature at the desired conditions; the controlled stage temperature can quickly be settled at its setpoint value. However, CS1 is not able to control the column pressure; the pressure is moved from the desired value (1 atm) to a new steady state (1.105 atm). This can be explained by the limitation of the condenser in which the maximum removal of heat is limited at 3500 kW. It can be seen that the condenser level increases to 72.01 % and the purity of BuOAc at the bottom decreases to 98.48 wt %. For case of 10% decrease in HOAc feed rate (Figure 5-3), both the temperature and pressure are controlled at the desired set point in 15 and 10 h, respectively. The BuOAc purity at bottom is 99.38 wt % at bottom rate of 38.12 kmol/h. However, the condenser level rises to 66.56 %.

Figure 5-4 shows that the responses of process are disturbed by  $\pm$  10 % BuOH feed rate. It is found that CS1 can handle with these changes. With 10 % increase of BuOH feed flow, product purity of 99.28 wt % and product rate of 42.38 kmol/h are obtained whereas with 10 % decrease of BuOH feed flow, the purity of BuOAc and the product flow rate are 99.56 wt % and 35.19 kmol/h, respectively. However, the condenser liquid level as  $\pm$  10 % BuOH feed rate is high.

Figures 5-5 shows the control response of CS1 for case of the step changes of feed composition (±5 wt % of HOAc and -5 wt % BuOH in fresh feed). It can be seen that when decreasing the concentration of HOAc from 80 wt % to 76 wt %, the results show that the purity of BuOAc of 99.30 wt % is obtained at the flow rate of 36.69 kmol/h. In this case, both of temperature and pressure can immediately be moved to the setpoint. However, when the concentration of HOAc in feed stream is changed

from 80 wt % to 84 wt %, CS1 is not able to maintain the column pressure due to the limited capability of the condenser. However, it can control the temperature at the setpoint. For case of a decrease in the concentration of BuOH (95 wt % BuOH in fresh feed), the results presented that CS1 cannot handle this disturbance; the system becomes unstable.

From the responses in Figure 5-3 to 5-5, it can be concluded that CS1 cannot only handle two disturbances (increasing the HOAc feed rate and changing BuOH feed composition). The condenser level is also high in all disturbances. Because of keeping temperature-controlled at setpoint, reboiler duty must be increased which lead to high vapor flow at the top of column. As the top column pressure is increased, condenser heat removal must be increased to maintain the top pressure, resulting in high liquid level in condenser. However, since the organic reflux flow is fixed, increased distillate rate is the one ways to maintain condenser liquid level. As a result, this control structure do not handled with the liquid level in reflux drum. To overcome such a difficulty, CS2 is proposed in order to rid of this problem.

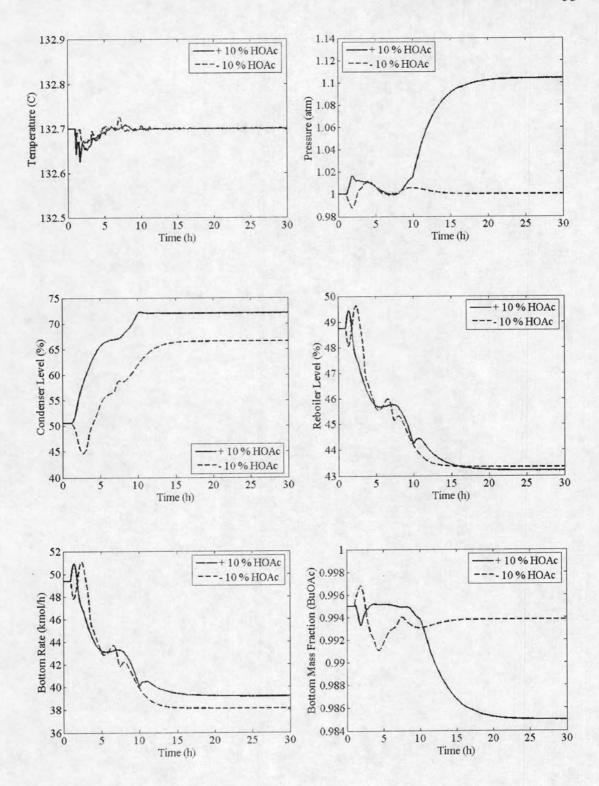


Figure 5-3 CS1 with  $\pm 10$  % HOAc feed rate.

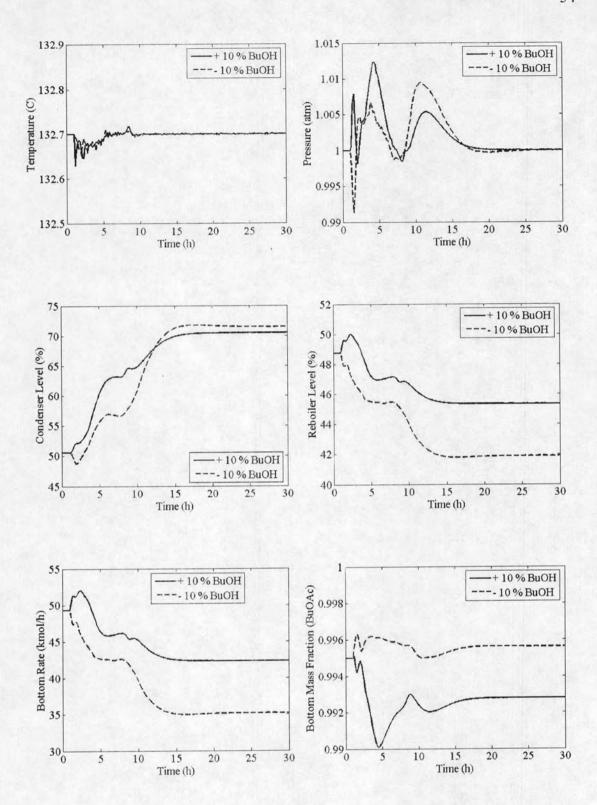


Figure 5-4 CS1 with  $\pm 10$  % BuOH feed rate.

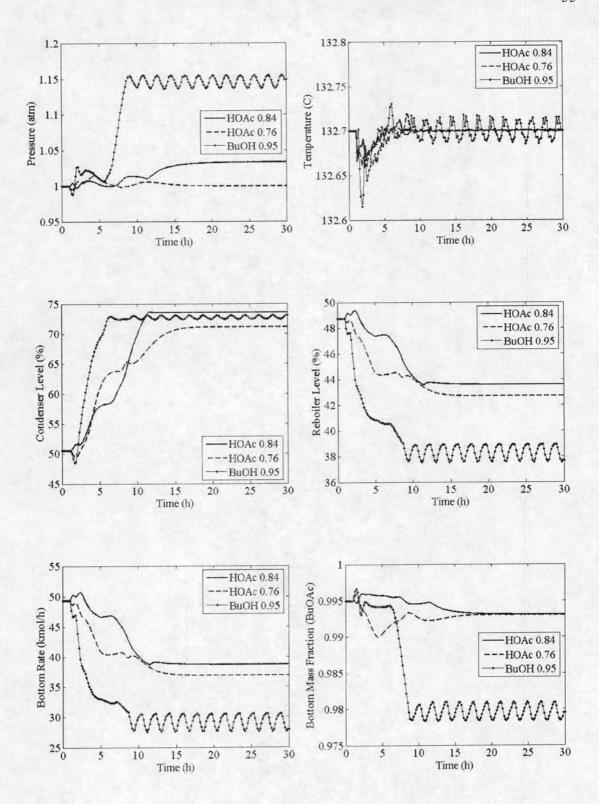


Figure 5-5 CS1 with step change in composition of feeds stream.

# 5.4 Control structure 2 (CS2)

In CS2, the fresh feeds of HOAc and BuOH are flow-controlled. The column pressure is controlled by manipulating the condenser heat removal. The base level is controlled by bottom flowrate. The product composition is indirectly controlled by the control of the temperature as in CS1. Figure 5-6 shows CS2 in which a temperature is controlled at stage 24<sup>th</sup> by manipulating reboiler duty. However, the difference between CS1 and CS2 are organic reflux flow and condenser level controlled-loops. The reflux ratio can be controlled at a desired value in CS2. The reflux drum level is controlled by manipulating the reflux, and a constant reflux ratio (equal to the steady state value) is maintained by adjusting the distillate flowrate. The tuning parameters of the controllers are shown in Table 5-2.

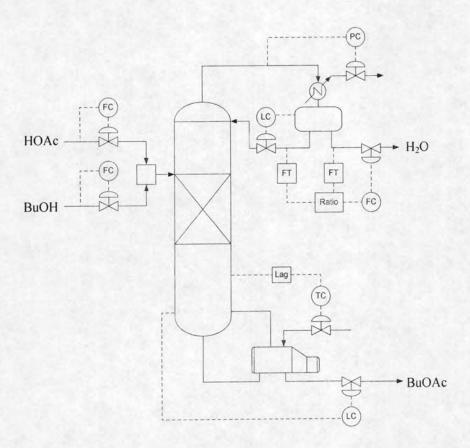


Figure 5-6 Control structure 2: constant reflux ratio.

Table 5-2 Tuning parameters for CS2

Temperature controller	$K_c = 2.15$
	$\tau_i = 2.67 \text{ min}$ $\tau_d = 0.593 \text{ min}$
Pressure controller	$K_c = 2$
Deflect describeral controllers	$\tau_i = 10 \text{ min}$ $K_c = 2.5$
Reflux drum level controllers	
Base level controller	$K_c = 2$
HOAc fresh feed controller	$K_c = 0.5$ $\tau_i = 0.3 \text{ min}$
BuOH fresh feed controller	$K_c = 0.5$
	$\tau_i = 0.3 \text{ min}$

## 5.4.1 Responses of Control Structure 2 (CS2)

In this section, the control performance of CS2 is evaluated under disturbance cases. Figures 5-7 to 5-8 show the control response when the flow rate of HOAc and BuOH are increased and decreased by 10% whereas Figure 5-9 demonstrate the response for the step changes in feed composition.

In Figure 5-7, when the process is disturbed in  $\pm$  10 % HOAc feed rate. The temperature and pressure controlled-loops are settled in around 15 and 20 h, respectively. While the liquid level in reflux drum is not higher than 50 %. However, it is observed that the BuOAc product purity is off spec (lower than 99.5 wt %).

When step changes in BuOH feed rate ( $\pm$  10 %) is introduced, the control responses are shown in Figure 5-8. The controlled stage temperature can quickly be settled in its setpoint value around 12 h. However, the pressure controlled-loop takes a long time to settle at 1 atm. The bottom products purity obtained is 99.21 and 99.54 wt % for + 10 % and – 10 % change in BuOH feed flowrate, respectively.

The control performance of CS2 for case of the step changes in feed composition are shown in Figure 5-9. The CS2 can handle with these changes. The temperature-controlled and the pressure-controlled are able to maintain at their desired setpoint and the condenser liquid level is not higher than 51 %. However, the product purity of BuOAc at bottom is still lower than the desired value.

From Figures 5-7 to 5-9, it can be seen that the CS2 is able to maintain the condenser level (not higher than 55 %). Due to reflux drum level is maintained by organic reflux rate, and organic reflux rate is manipulated distillate flow (reflux ratio is constant). Importantly, CS2 is able to maintain the temperature and pressure at desired set point in all simulations. It should be noted that although CS2 can accurately control the temperature of 24<sup>th</sup> stage, the product composition is changed from the design value. This indicates the limitation of CS2. Therefore, CS3 is designed for the control of reactive distillation with respect to the BuOAc purity at 99.5 wt % as a desired target.

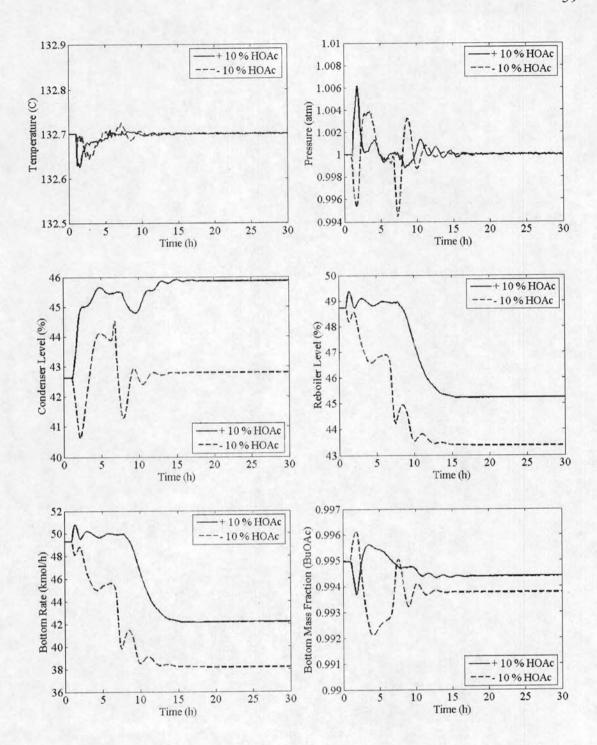


Figure 5-7 CS2 with  $\pm 10$  % HOAc feed rate.

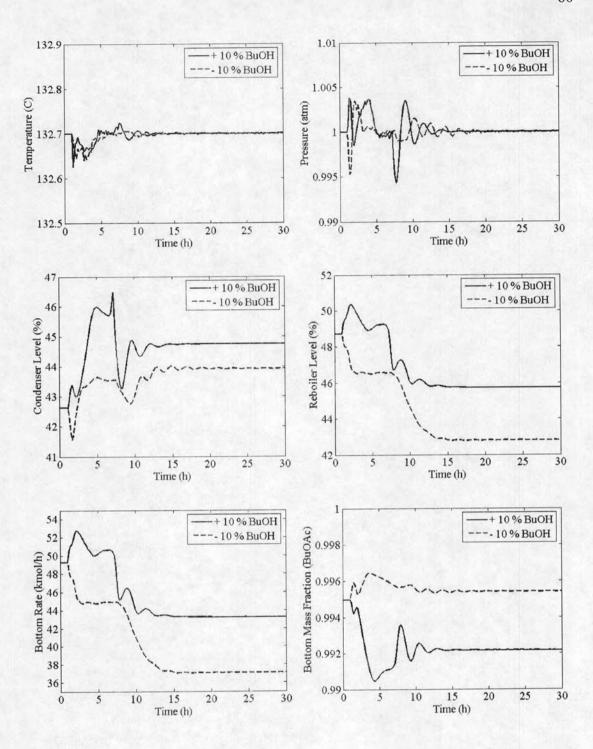


Figure 5-8 CS2 with  $\pm 10$  % BuOH feed rate.

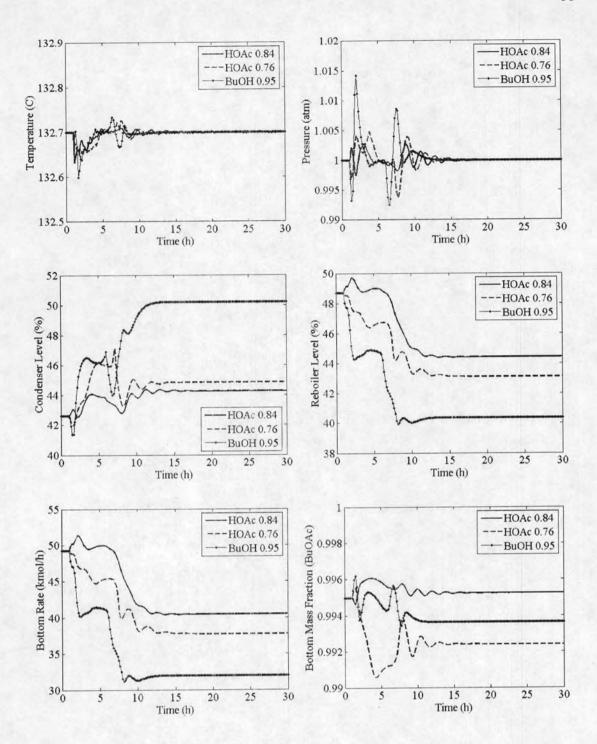


Figure 5-9 CS2 with step change in composition of feeds stream.

# 5.5 Control structure 3 (CS3)

Figure 5-10 shows the control structure using this strategy. The most of the controlled-loops in CS3 are same as CS2. Nevertheless, instead of using indirectly composition control, the directly composition control is selected. It is noted that the bottom product purity is controlled by manipulating the vapor boilup. The tuning parameters for CS3 are shown in Tables 5-3.

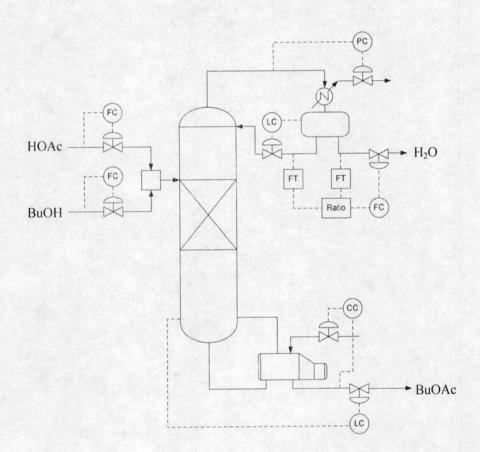


Figure 5-10 Control structure 3: direct product controlled.

Table 5-3 Tuning parameters for CS3

Composition controller	$K_c = 4$ $\tau_i = 25 \text{ min}$
Pressure controller	$K_c = 2.5$ $\tau_i = 15 \text{ min}$
Reflux drum level controllers	$K_c = 2.2$
Base level controllers	$K_c = 2$
HOAc fresh feed controller	$K_c = 0.5$ $\tau_i = 0.3 \text{ min}$
BuOH fresh feed controller	$K_c = 0.5$ $\tau_i = 0.3 \text{ min}$

## 5.5.1 Responses of Control Structure 3 (CS3)

As mentioned earlier, in CS3, the purity of the bottom product is directly controlled by manipulating the reboiler heat duty. The column pressure is controlled by the removal of heat at condenser. The reflux drum level is controlled by manipulating the reflux, and a constant reflux ratio (equal to the steady state value) is maintained by adjusting the distillate flowrate. The fresh feeds of HOAc and BuOH are flow-controlled.

In this section, the control performance of CS3 is analyzed under the case of disturbance rejection. Figures 5-11 and 5-12 show the control response when the flow rate of HOAc and BuOH are increased and decreased by 10% whereas Figure 5-13 demonstrate the response for the step changes in feeds composition. It can be seen that the CS3 is able to maintain the composition and pressure at the desired set point in all simulations. But the responses of the process have a swing too much in 5 h and the swings of the responses decreased in 15 h. The dynamics of the scheme are slower than the others because of the lag in the bottom composition to reboiler duty loop. Further, the composition of bottom product is regulated at the desired steady-state value.

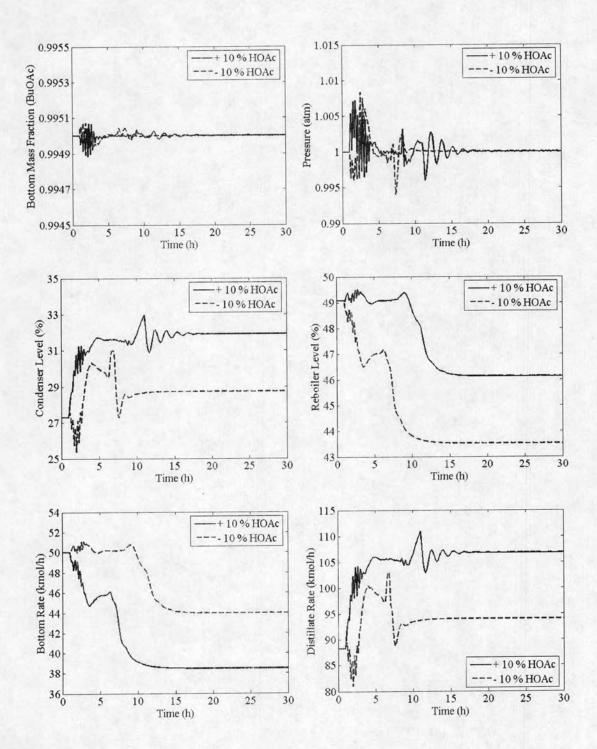


Figure 5-11 CS3 with  $\pm 10$  % HOAc feed rate.

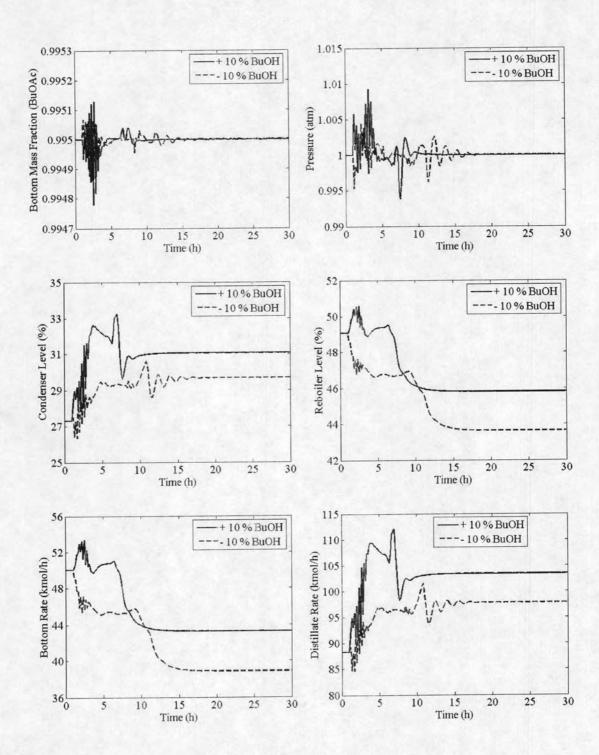


Figure 5-12 CS3 with  $\pm 10$  % BuOH feed rate.

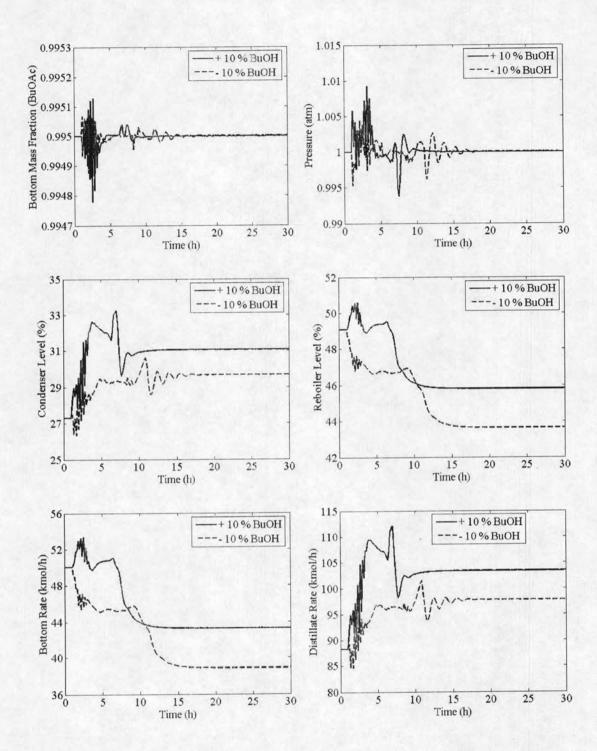


Figure 5-13 CS3 with step change in composition of feeds stream.